IS 6770 (1984): Benzophenone [PCD 18: Natural and Synthetic Fragrance Materials]

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IS : 6770 - 1984
(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR BENZOPHENONE

(First Revision)

UDC 665·574 : 547·631·6

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

November 1984

Indian Standard

SPECIFICATION FOR BENZOPHENONE

(First Revision)

Natural and Synthetic Perfumery Materials
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Indian Standard

SPECIFICATION FOR BENZOPHENONE

(First Revision)

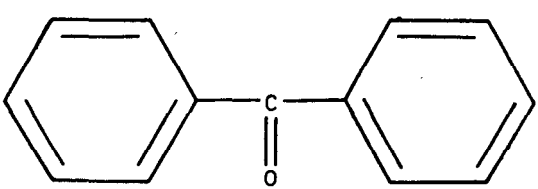
0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 16 August 1984, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1972. The Sectional Committee responsible for its preparation felt that the standard should be revised with a view to bring it in line with trade practices in perfumery technology and also align it with the quality level of the material currently manufactured and sold in the country.

0.3 In the present revision the requirment for melting point has been introduced to check the quality of the material. Gas chromatographic analysis has also been included as it is being progressively used in the country.

0.4 Benzophenone (diphenyl ketone, benzoyl benzene) (C_{13} $H_{10}O$) has not been found in nature. It is synthetically prepared by the Friedel and Crafts' reaction. It is a white material having a sweet and mild rose geranium like odour. It is represented by the following structural formula:



BENZOPHENONE (Molecular Mass 182·1)

0.5 Benzophenone is widely used in various types of perfumes and occasionally in flavours. It also acts as a good fixative. Soap perfumes generally contain this where it acts as an antioxidant. Benzophenone is used as an intermediate in pharmaceuticals and other organic synthesis and some of its derivatives are used as ultraviolet light absorbers.

0.6 In the preparation of this standard, considerable assistance has been derived from the following publications:

EOA No. 83, 1975 Standard for benzophenone, published by the Essential Oil Association of USA, New York.

The Givaudan Index, 1978, published by Givaudan-Delawanna Inc, New York.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for benzophenone.

2. TERMINOLOGY

2.1 For the purpase of this standard, definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description — The material shall consist of white rhombic crystals or powder, free from foreign matter and contaminants.

3.2 Solubility — One gram of the material shall be clearly soluble in 7 volumes of 95 percent ethanol and 10 volumes of 80 percent ethanol, when tested as prescribed in **8** of IS : 326-1968‡.

3.2.1 Benzophenone is soluble in diethyl phthalate, benzyl benzoate and many fixed or mineral oils, whereas it is insoluble in water and glycerine.

3.3 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in fibre containers or suitably lined cans.

4.2 Marking — Each container shall bear legibly or indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and recognized trade-mark, if any;
c) Net mass of the material; and
d) Batch number.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

TABLE 1 REQUIREMENTS FOR BENZOPHENONE

(*Clauses* 3.3, 5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Odour	Persistent, rosy odour	IS : 2284-1963*	—
ii)	Freezing point, °C, *Min*	47	IS: 326 (Part 18)-1984†	—
iii)	Melting point, °C, *Min*	48	**16.8** of IS : 326-1968‡	—
iv)	Freedom from chlorine	To pass the test	**16.10** of IS : 326-1968‡	—
v)	Benzophenone content, percent by mass, *Min*	99	—	A

*Method for olfactory assessment of natural and synthetic perfumery materials.

†Methods of sampling and test for natural and synthetic perfumery materials : Part 18 Determination of freezing point (*second revision*).

‡Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

5.2 Number of Tests

5.2.1 Benzophenone content shall be tested on each of the individual samples.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

5.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity — The lot shall be declared as conforming to the requirements of the specification if **5.3.1** and **5.3.2** are satisfied.

5.3.1 For benzophenone centent, the mean $\overline{X}$ and range (R) of test results shall be calculated as follows:

$$\text{Mean } (\overline{X}) = \frac{\text{Sum of the test results}}{\text{Number of the test results}}$$

Range (R) = Difference in the maximum and minimum of the test results

The lot shall be deemed to a have satisfied the requirement for this characteristic if the value of the expression $\overline{X} - 0{\cdot}6\ R$ is greater than or equal to the minimum limit for benzophenone content given in Table 1.

5.3.2 All the test results on the composite sample meet the relevant specification requirements.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed under **3.1**, **3.2** and the appropriate references specified under col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (v)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF BENZOPHENONE CONTENT

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the

*Specification for water for general laboratory use (*second revision*).

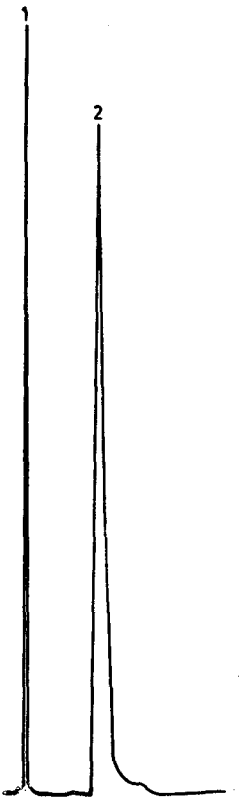
constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and one detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being separated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for benzophenone using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Sample	Benzophenone
Column	
a) Material	Copper
b) Length	1·83 m
c) O.D	0·635 cm
d) I.D	0·476 cm
e) Stationary phase	Carbowax 20 M, 10 percent by mass
f) Solid support	Chromosorb WAW 60-80 mesh
Carrier Gas	Nitrogen
Conditions	
a) Column temperature isothermal	205°C
b) Injection port temperature	200°C
c) Carrier gas flow rate	50 ml/min
d) Inlet pressure	3·5 kg/cm^2
Detector	
a) Type	F.I.D. (Flame ionisation detector)
b) Temperature	280°C
Recorder	
a) Span	IMV
b) Chart speed	0·25 cm/min
Attenuation	64 (Range : 100)

NOTE — The analysis may also be accomplished with columns containing : DEGS (Diethylene glycol succinate) and FFAP (Free fatty acid phase).



1 — Solvent
2 — Benzophenone

FIG. 1 TYPICAL CHROMATOGRAM OF BENZOPHENONE

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample (dissolved in the suitable solvent) at injection part. Where it is vapourized and well mixed with the carrier gas. This is led into the chromatographic column wherein vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding with amount of particular constituent leaving the column. The detector signals, on transmission with recorder, plots in chart. From

the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

A-3. CALCULATION

A-3.1 Area Measurements (*see* **Note 1**) — Since normal peaks approximate a triangle the area is measured by multiplying the peak height times the width of the half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.2 Area Normalization (*see* **Note 2**) — By normalizing, it is meant calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurements, namely Triangulation, Disc integrator and Electronic Digital Integrator if fixed with GLC machine would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

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